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# The Effects of Pigeage, Délestage, Remontage and Oxygenation Treatments Applied during Maceration on Phenolic Content, Aroma Composition and Sensory Properties of Red Teran (*Vitis vinifera* L.) Wine

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**Abstract:** The aim of this study was to evaluate the effects of mechanical (pigeage, délestage and remontage) and oxygenation treatments on the phenolic and aromatic compounds and sensory characteristics of Teran wines. The experiment included a 20-day maceration period, during which the above-mentioned treatments were applied, as well as the post-fermentation processes of pressing and first rack. The analysis of phenolic, chromatic and aroma compounds and the sensory characterization of the wines were used to describe the effects of the treatments investigated. After the observed maceration period, remontage resulted in wines with the highest total phenols (2682.0 ± 14.8 mg GAE/L). In contrast, délestage resulted in the lowest total phenols (2499.1 ± 17.6 mg GAE/L) and total anthocyanins (530.1 ± 2.8 mg/L) and had the strongest effects on chromatic characteristics. The post-fermentation processes (pressing, racking) showed similar trends and resulted in higher phenolic concentrations in the remontage wine, while the délestage was again characterized by lower total phenol and anthocyanin concentrations. In addition, the délestage wine contained a higher concentration of almost all analyzed esters and two higher alcohols (2-methylpropan-1-ol and 1-hexanol), while the remontage wine had the highest concentration of 2-phenylethanol and 3-methylbutyl acetate. Finally, maceration proved to be a key factor in defining the wines' sensory characteristics, with the remontage-treated wine showing the best overall quality.

**Keywords:** Teran wine; maceration treatments; phenolics; aroma; color



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## 1. Introduction

Color and phenolic and aromatic compounds are the most important characteristics of grapes and wine when it comes to defining their quality. Their concentration and composition are influenced by various factors such as the growing region, grape variety, vintage, terroir and production technique [1]. Among the winemaking processes, maceration plays a key role in determining red wine's quality. In particular, maceration influences the sensory characteristics of wines, such as the color, taste and flavor, with phenolic and aromatic compounds being extracted from the skins and seeds to varying degrees, regardless of the grape variety [2].

In conventional red wine production, maceration usually takes place during the short pre-fermentative phase and during alcoholic fermentation, after which pressing is commonly conducted. However, the contact of skins and seeds can be extended from a few days to several weeks or months [3]. Several studies have shown that prolonged maceration can improve the extraction of phenolic compounds [4–7], stabilize the wine color [8,9] and

influence certain sensory characteristics of the wine [6,10–12]. Moreover, the efficiency of extraction also depends on the mechanical treatments applied. For example, punch downs, pump overs or racking have been used to accelerate the extraction and diffusion of valuable compounds from different grapes into the must, resulting in a higher concentration of phenolic compounds and an improved sensory quality of the wines produced [11,13–15]. In the punch down treatment, also known as pigeage, the cap of pomace is immersed in the must by vertical pressure. In pumping over or remontage, the fermenting must is transferred from the bottom to the top of the tank, while in délestage or rack and return, all the must is transferred from one tank to another, and the liquid is then pumped back into the first tank and over the pomace [16].

In addition, grape must and wine are spontaneously or intentionally exposed to different oxygen concentrations during vinification. In this context, oxygenation usually means the intentional and controlled exposure of must/wine to oxygen, with the aim of improving the overall wine quality [17]. Factors that influence the positive or negative outcome of oxygenation are the time of application, the amount of oxygen added and the phenolic characteristics of the wine [18]. For example, it has been reported that the addition of oxygen to the must in combination with some mechanical processes during maceration favors the extraction of phenolic compounds in red wine production [19]. There is a lack of comparative studies in the literature examining different treatments on the same variety and vintage. Moreover, few studies address the effects of maceration treatments on the composition of phenolic and aromatic compounds [11,13,14], which are inextricably linked, on the sensory and overall quality of the wine.

The objective of this study was to evaluate the effects of different maceration techniques such as pigeage, délestage, remontage and oxygenation together with prolonged maceration on the chemical composition and sensory characteristics of Teran red wine. The production of this wine, which is the predominant red grape variety in Istria, Croatia, usually involves 5–10 days of skin contact [20], while prolonged maceration in combination with different cap management methodologies has not yet been studied. The results obtained will contribute to a better understanding of the effects of maceration techniques on the overall quality of red wines in general.

## 2. Materials and Methods

### *2.1. Materials*

#### 2.1.1. Chemicals

Ethanol was HPLC-grade and purchased from J.T. Baker (Deventer, The Netherlands), while sodium chloride p. a. and hydrochloric acid (37%) were purchased from Carlo Erba reagents GmbH (Emmendingen, Germany). Folin–Ciocalteu reagent was obtained from Kemika (Zagreb, Croatia), sodium bisulfite from Acros Organics (Geel, Belgium) and sodium carbonate anhydrous from T.T.T. Sveta Nedelja, Croatia. All standards were purchased from Sigma-Aldrich (St. Louis, MO, USA), inlcuding gallic acid and standards of individual aroma compounds. Deionized water was purified with the Milli-Q water system (Millipore Corp., Bedford, MA, USA).

#### 2.1.2. Grapes

The red grapes of *Vitis vinifera* L. cv. Teran were manually harvested in September 2022 in Motovun (Central Istria, Croatia) at technological maturity (reducing sugars 225 ± 3.1 g/L; pH 3.15 ± 0.0; total acidity 8.9 ± 0.1 g/L as tartaric acid) and transported to the experimental winery of the Department of Agriculture of the Rijeka Polytechnic in Poreč, Croatia.

#### 2.1.3. Winemaking and Maceration Treatments

A quantity of 1074 kg of Teran grapes was destemmed, crushed, sulfited (potassium metabisulfite at a concentration of 10 g/hL) and homogenized to reduce possible differences in the composition of the crushed grapes' composition. The homogenized must with skins

and seeds was then evenly distributed among eight stainless steel tanks (130 L each) and underwent spontaneous alcoholic fermentation and maceration (with the treatments mentioned below). On the third and sixth day of alcoholic fermentation, the yeast nutrient Fermaid E (Lallemand, Germany) was added at a concentration of 15 g/hL in two equal portions. The fermentation temperature was maintained at 25 °C for all variants, and sugar consumption was monitored twice daily by recording the soluble solids (hydrometer, °Brix).

Maceration was carried out in two phases: fermentative maceration (first 10 days) and prolonged maceration (further 14 days), with four different treatments:

(i) Pigeage (control treatment), in which the caps were punched down with a stainless steel stick with a flat plate end ('*pigeou*') two times per day, with an interval of 10–12 h between immersions;

(ii) The délestage treatment involved a rack and return procedure of 30–40 L of fermenting must/wine on the 3rd, 5th, 7th, 12th and 14th days, together with punching down the cap twice a day (the racked fermenting must/wine was kept in a separate stainless steel tank for one hour before being returned to the original tank);

(iii) The remontage treatment consisted of 12 min of pumping over operations on the 3rd, 5th, 7th, 12th and 14th day, along with punching down the cap twice a day;

(iv) The oxygenation treatment consisted of a total of 40 mg/L of oxygen added in four portions (3rd day: 5 mg/L, 5th: day 10 mg/L, 7th: day 15 mg/L, and 18th day: 10 mg/L), with the punch down treatments performed daily as in the previous variants. Oxygen was supplied using a silicone diffuser located at the bottom of the tank, and the oxygen flow controller was used to measure the oxygen supply, as described by Lukić et al. [21].

Each variation was given 24 days of contact time (fermentative and prolonged maceration) and was then pressed with a Lancman VSPIX 120 hydropress (Gomark, Vransko, Slovenia) at a maximum pressure of 0.4 bar. Immediately after pressing, the wines were sulfited to adjust the concentration of free $SO_2$ at 30 mg/L. Four days after pressing, each wine was racked, and the free $SO_2$ content was again adjusted to 30 mg/L. No malolactic fermentation was carried out on the wines obtained. Samples of each treatment were taken after 3, 5, 7, 12, 14, 18 and 20 days of maceration, as well as after pressing and racking (as shown in Figure 1). Samples were frozen (−20 °C) immediately after collection and stored in the freezer until the laboratory analysis. The average composition of the Teran wines produced was as follows: alcoholic strength 14.0 ± 0.2% (*v*/*v*), pH 3.2 ± 0.0 and total acidity 8.2 ± 0.2 g/L as tartaric acid, with no statistical differences between treatments.

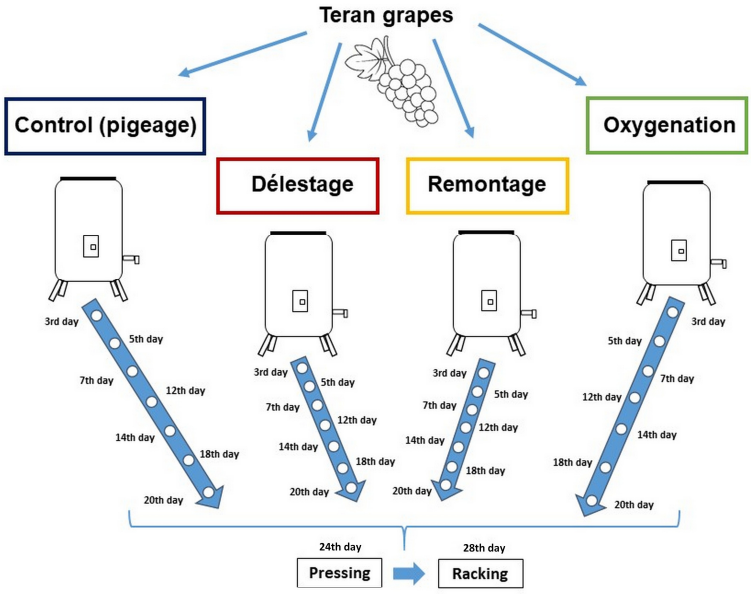


**Figure 1.** Production scheme and sampling points of Teran wines.

### *2.2. Methods*

#### 2.2.1. Spectrophotometric Analysis of Phenolic Compounds

Total phenolics (TPs) were determined by the Folin–Ciocalteu method according to Singleton and Rossi [22], and the results were expressed as gallic acid equivalents (mg GAE/L). Total anthocyanins (TAs) were analyzed using the bisulfite bleaching method described in detail by Ribéreau-Gayon and Stonestreet [23], and results were expressed as mg/L. All analyses were performed using the Specord 50 Plus spectrophotometer (AnalytikJena, Jena, Germany).

#### 2.2.2. CIELab Analysis of Chromatic Characteristics

The measurements of chromatic characteristics were conducted using the CIELab assay according to the published method OIV-MA-AS2-11 [24]. The CIELab parameters, including L* (lightness), a* (redness/greenness), b* (yellowness/blueness), C* (chroma) and H* (hue angle), were determined. The total color difference ($\Delta E^*_{ab}$) was then calculated using the following equation:

$$\Delta E^*_{ab} = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (1)$$

#### 2.2.3. GC/MS Analysis of Aroma Compounds

The aroma compounds were analyzed by solid phase microextraction (SPME) in combination with gas chromatography coupled with mass spectrometry (GC/MS) according to a previously published method [25]. An Agilent 6890 series Gas Chromatography system with an Agilent 5973 Inert mass selective detector (Agilent Technologies, Santa Clara, CA, USA) was used. For the SPME technique, 10 mL of the sample was mixed with the internal standard 1-pentanol (20 mg/L) and 2 g of sodium chloride in a 20 mL vial using a magnetic stirrer. The vial was sealed with a PTFE silicone septum, and a 100 µm PDMS fiber (Supelco, Bellefonte, PA, USA) was inserted into the headspace of the vial to extract the aroma compounds for 30 min at 40 °C under constant agitation. The fiber was then transferred to the GC injector to desorb the compounds at 250 °C for 5 min. A BP20 capillary column (50 m × 220 µm id, 0.25 µm film thickness, SGE Analytical Science, Victoria, Australia) was used to separate the aroma compounds, with the following temperature conditions: 40 °C for 5 min, then increasing to 200 °C at a rate of 3 °C/min, further increasing from 200 to 240 °C at a rate of 30 °C/min, and holding at 240 °C for 1 min. The full scan mode from $m/z$ 30 to 330 was used to detect the compounds. Helium was used as carrier gas with a flow rate of 1.2 mL/min in splitless mode. The ion source in EI mode operated at 70 eV and maintained at 150 °C, while the detector interface temperature was set to 250 °C. The identification of the analyzed aroma compounds was performed using MSD Chemstation software (version G1701CA C.00.01, Agilent Technologies, Santa Clara, CA, USA). For quantification, a calibration curve was created for each compound and analyzed under the same extraction and chromatography conditions as for the samples.

#### 2.2.4. Sensory Analysis

The sensory analysis of the wines produced was carried out using two sensory methods, namely, the 100-point OIV method [26] and quantitative descriptive analysis (QDA) [27]. The sensory panel consisted of seven experienced wine tasters. All of the panelists were selected from the staff of the Agricultural Department of the Polytechnic of Rijeka. Before the formal sensory analysis, the panelists were tested in recognizing the basic flavors in a standard solution (sensitivity test) and detecting differences in the flavor of the red wine samples compared to the control (discriminant capability test) [28]. The training sessions were then carried out over three consecutive weeks (2 × 2 h sessions per week). During training, a total of 13 descriptors (cherry, sour cherry, raspberry, blackberry, blueberry, redcurrant, blackcurrant, plum, compote (sweet notes), spicy/herbs, forest fruit, astringency, body) were generated by developing a descriptor list of descriptors that are suitable for Teran wine [29]. Formal testing was conducted in separate booths, and each

sample was served in random order. Samples (35 mL) were presented in clear wine tasting glasses [30] and labeled with a three-digit code at room temperature (18–20 °C). During the QDA, judges were asked to couple each descriptor with a scale from 0 to 5 (0—absence of perception, 1—low, 2—slight, 3—moderate, 4—intensive, 5—very intensive). Prior to the 100-point method, a reference Teran wine that had already been assessed and classified as high-quality was evaluated in order to attune the judges' criteria.

#### 2.2.5. Statistical Analysis

A one-way analysis of variance (ANOVA) was performed using Statistica V.10 software (Statsoft Inc., Tulsa, OK, USA) to determine the significant difference between the means of the chemical composition data. A Tukey's HSD test was performed when significant differences ($p < 0.05$) were found between samples. Data are presented as the mean of four analytical repetitions with standard deviation.

## 3. Results and Discussion

### *3.1. Changes in Phenolics and Chromatic Characteristics during Maceration*

The effects of different mechanical treatments (pigeage, délestage and remontage) and oxygenation during maceration on the total phenolics (TPs), total anthocyanins (TAs) and chroracteristics (L*, a*, b*, C* and H*) are shown in Table 1 and Figure 2. In agreement with previous observations [6,7,11,31], an increase in TP concentration was observed throughout the duration of the prolonged maceration until the 18th day. After 7 days of maceration, some initial trends disappeared, and there were no significant differences between treatments after 12 and 14 days (Table 1). However, as the maceration time progressed further, new trends appeared, resulting in the highest TP concentrations in the remontage sample after 20 days compared to the other three treatments ($p < 0.05$). In addition, no significant differences were found between pigeage (control) and oxygenated wines, while délestage resulted in the lowest concentrations of TPs. Several studies have investigated different maceration treatments (délestage, saignée, punch down, pump over, prolonged maceration) to increase the extraction of phenolics during the fermentation of red wines [14,15,32–34]. De Beer et al. [33] found that the punch down treatment (pigeage) resulted in Pinotage wines with higher phenolic contents and higher total antioxidant capacities compared to the pump over treatment (remontage). This was due to the milder conditions of the pump over treatment, but also to the possibility of greater oxygen exposure when performing the treatment compared to punch down, which leads to polymerization and precipitation of phenolics, reducing their concentration [14]. However, as the remontage treatment in our case involved a more intensive mechanical approach with both pumping over and punching down, the trends obtained were expected (Table 1). In addition, the final effect of the above procedures strongly depends on the variety, vintage, ripeness of the grapes and winemaking style [3,13,35]. In agreement with our results, Soto Vázquez et al. [34] reported that there is no difference in the extraction of phenolic compounds between six winemaking techniques (conventional maceration, pre-fermentative maceration, délestage, Ganimede fermentation system, additions of enzymes and tannins, addition of oak chip) after alcoholic fermentation of red Mencía wine.

The TA concentrations increased rapidly during the first 7 days and then decreased (Table 1). These results are in agreement with previous studies, in which the maximum anthocyanin concentration was reached during the first 7 days [3,6,36], while a further decrease during a prolonged maceration was accompanied with a higher extraction of tannins [12,37,38]. Indeed, the extraction rate of anthocyanins decreases when an equilibrium based on an adsorption/desorption mechanism is reached between the concentration of anthocyanins in grapes and wines [38]. Moreover, anthocyanins are a rather unstable species that undergoes various chemical reactions during winemaking, including degradation reactions, the incorporation of anthocyanins into polymeric pigments and the formation of pyranoanthocyanins [8,12,39]. The formation of polymeric pigments is primarily the result of condensation reactions between anthocyanins and tannins [39], where anthocyanins

can react either as nucleophiles undergoing electrophilic aromatic substitution on the A ring, or as electrophiles undergoing nucleophilic addition on the central C ring [40]. In addition, the formation of pyranoanthocyanins is also related to the loss of anthocyanins during maceration in the fermenting must [41]. Their formation in the first stage of red winemaking is the result of reactions between anthocyanins and some metabolites (pyruvic acid, acetoacetic acid and acetaldehyde) that are released during yeast fermentation (type A vitisins) and reactions between pyruvic acid and anthocyanins (type B vitisins) [42]. The results obtained (Table 1) showed that the concentrations of TA extracted on day 7 followed the order of remontage, pigeage, délestage and oxygenation ($p < 0.05$). However, the TA loss was more pronounced during délestage, resulting in significantly lower TA concentrations in this wine compared to the others ($p < 0.05$). This could be due to the initially lower extraction rate for the délestage wine during the first days of fermentation (Table 1), as well as the intensive formation of polymeric pigments that are known to be favored by this treatment [9,32].

**Table 1.** Changes in total phenolics (TPs) and total anthocyanins (TAs) of fermenting musts/wines during 20 days of maceration for the studied winemaking treatments.

| Days | Pigeage (C) | Délestage | Remontage | Oxygenation |
|---|---|---|---|---|
| | | TP (mg GAE/L) | | |
| 3 | 1418.6 ± 7.5 $^{c}$ | 1711.8 ± 18.0 $^{b}$ | 1772.8 ± 18.4 $^{ab}$ | 1802.3 ± 20.5 $^{a}$ |
| 5 | 2046.4 ± 19.0 $^{a}$ | 1964.5 ± 20.9 $^{a}$ | 1986.8 ± 22.1 $^{a}$ | 1863.2 ± 20.4 $^{b}$ |
| 7 | 2092.5 ± 17.7 $^{ab}$ | 2028.2 ± 21.1 $^{bc}$ | 2127.9 ± 13.0 $^{a}$ | 2000.5 ± 27.9 $^{c}$ |
| 12 | 2301.8 ± 24.9 $^{a}$ | 2231.0 ± 21.1 $^{a}$ | 2279.0 ± 20.1 $^{a}$ | 2200.5 ± 25.7 $^{a}$ |
| 14 | 2532.0 ± 27.2 $^{a}$ | 2442.6 ± 16.0 $^{a}$ | 2540.8 ± 10.9 $^{a}$ | 2520.6 ± 36.6 $^{a}$ |
| 18 | 2633.3 ± 26.5 $^{ab}$ | 2539.7 ± 27.2 $^{b}$ | 2690.9 ± 20.8 $^{a}$ | 2662.6 ± 28.6 $^{a}$ |
| 20 | 2609.6 ± 16.6 $^{b}$ | 2499.1 ± 17.6 $^{c}$ | 2682.0 ± 14.8 $^{a}$ | 2615.6 ± 12.8 $^{b}$ |
| | | TA (mg/L) | | |
| 3 | 556.0 ± 2.9 $^{b}$ | 722.8 ± 2.9 $^{a}$ | 556.2 ± 4.1 $^{b}$ | 400.9 ± 2.0 $^{c}$ |
| 5 | 724.7 ± 1.7 $^{b}$ | 767.1 ± 0.8 $^{a}$ | 710.9 ± 1.9 $^{c}$ | 712.5 ± 2.0 $^{c}$ |
| 7 | 806.4 ± 3.8 $^{b}$ | 781.9 ± 4.4 $^{c}$ | 870.5 ± 4.0 $^{a}$ | 735.3 ± 4.6 $^{d}$ |
| 12 | 757.0 ± 2.4 $^{b}$ | 610.5 ± 3.2 $^{d}$ | 770.0 ± 3.3 $^{a}$ | 718.3 ± 3.2 $^{c}$ |
| 14 | 742.1 ± 3.7 $^{a}$ | 580.5 ± 3.8 $^{c}$ | 740.3 ± 5.1 $^{a}$ | 707.0 ± 1.4 $^{b}$ |
| 18 | 683.0 ± 2.3 $^{a}$ | 547.2 ± 3.4 $^{c}$ | 691.1 ± 4.6 $^{a}$ | 660.3 ± 2.2 $^{b}$ |
| 20 | 628.9 ± 4.1 $^{a}$ | 530.1 ± 2.8 $^{b}$ | 625.2 ± 3.3 $^{a}$ | 624.7 ± 2.9 $^{a}$ |

Data presented as mean value of four repetitions ± standard deviation as error bars. ANOVA to compare data; different letters indicate statistical differences between musts/wines of all treatments at the same time (Tukey's test, $p < 0.05$).

Similarities were observed between the treatments with regard to the evolution of chromatic parameters, as shown in Figure 2a–e. During the first 7 days, there was a decrease in L*, b* and H* values and an increase in C* and a* in all treatments. After this first period, the L* values showed a tendency to increase, as did the H* and b* values, but to a lesser extent, while the C* and especially the a* values tended to decrease. These results show that anthocyanin extraction, which peaks in the first 7 days, corresponds to the highest C* and a* (highest color and redness) and the lowest L*, b* and H* values (less light and less orange hues). The changes observed in the chromatic parameters were consistent with previously observed changes in the concentrations of total anthocyanins during maceration (Table 1) and with data from the literature [43,44].

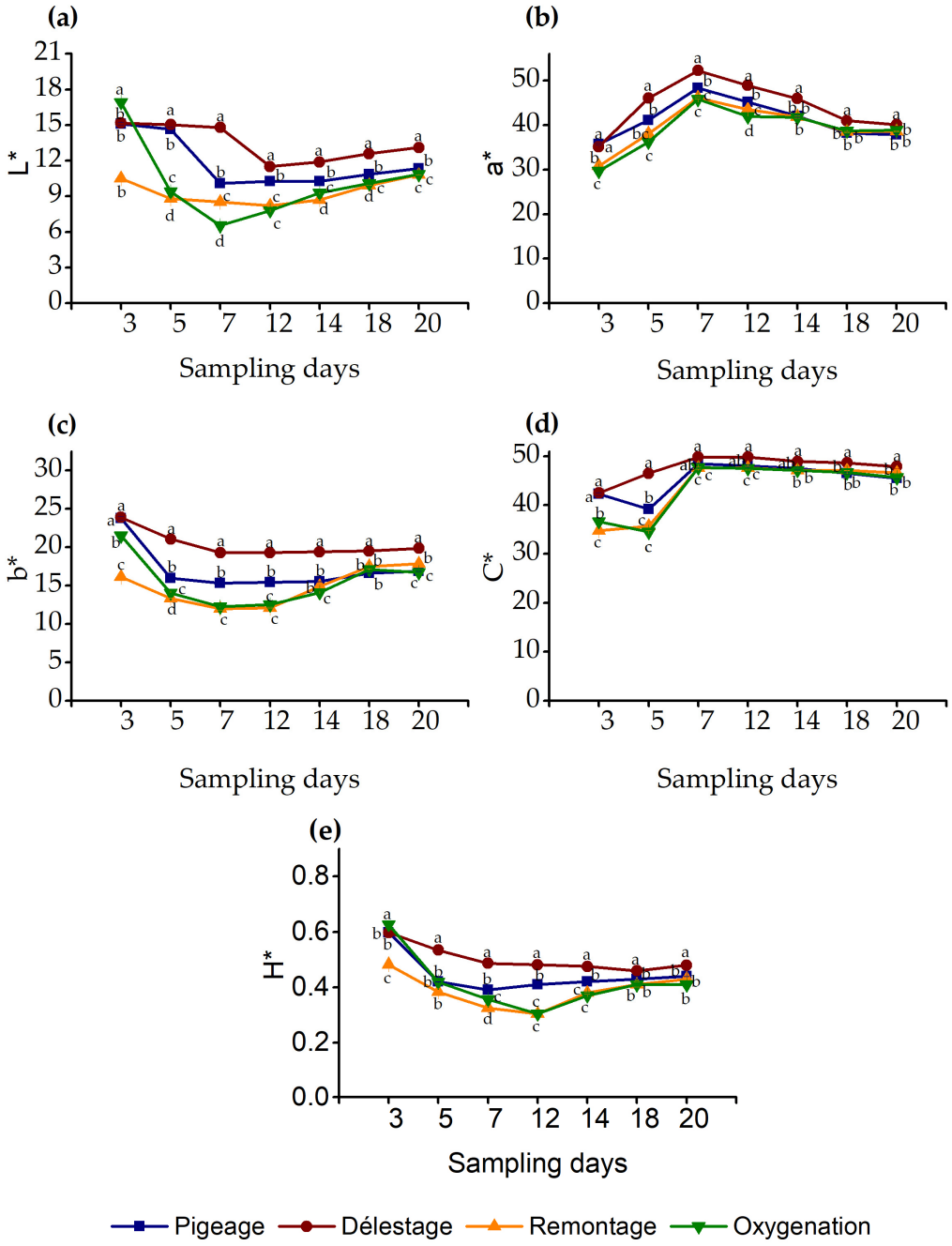


**Figure 2.** Changes in chromatic characteristics: (**a**) L*, (**b**) a*, (**c**) b*, (**d**) C*, (**e**) H* of fermenting musts/wines during 20 days of maceration for studied winemaking treatments. Data presented as mean value of four repetitions ± standard deviation as error bars. ANOVA to compare data; different letters indicate statistical differences between musts/wines of all treatments at same sampling point (Tukey's test, $p < 0.05$).

Anthocyanins are primarily responsible for the color of young red wine [31,36], and once extracted from the skins into the wine, they are involved in various chemical reactions. They can react with tannins to form oligomeric and polymeric pigments, form pyranoanthocyanins or undergo transformation and degradation reactions depending on the conditions of the medium. This leads to a different color intensity, as well as a higher hue (red-orange tones) [41] and a higher stability [43,44]. In addition, the effects of different maceration treatments on chromatic characteristics have been shown to be vary widely [9,34,45] depending on the phenolic profile of the wine and the winemaking conditions applied. In

the present study, no significant differences in chromatic characteristics were observed between all three treatments (pigeage, délestage and remontage) after 20 days of maceration. However, the délestage treatment showed the most significant impact on the chromatic characteristics of the wine among all other treatments, which were maintained through the prolonged maceration period. This wine had the significantly highest L*, b* and H* values, as well as C* and a* values ($p < 0.05$). Thus, a brighter wine, with orange and red hues was produced, but with greater chromatic purity related to higher C* values, possibly due to less mixture of pigments [34]. These results could be attributed to the lower concentrations of TA that were extracted during maceration, but also to the formation of polymeric pigments and/or pyranoathocyanins, which are known to be favored in a slightly oxygenated environment [9,40].

### *3.2. Changes in Aroma Composition during Maceration*

The aroma compounds identified and quantified in the fermenting musts/wines during the 20-day maceration are shown in Figure 3. These compounds belong to the quantitatively most important groups of aroma compounds in wine: esters (ethyl acetate, 3-methylbutyl acetate, 2-phenylethyl acetate, ethyl hexanoate, ethyl octanoate, ethyl decanoate and diethyl butanedioate) and higher alcohols (2-methylpropan-1-ol, 3-methylbutan-1-ol, 1-hexanol and 2-phenylethanol). Regarding the esters analyzed (Figure 3A–G), there were no significant differences between the treatments applied after 20 days of maceration, with the exception of diethyl butanedioate, for which the délestage resulted in a higher concentration compared to the other treatments studied (Figure 3G). Among the esters, only ethyl acetate showed a constant increase during the 20-day maceration period. On the other hand, 3-methylbutyl acetate, 2-phenylethyl acetate, ethyl hexanoate, ethyl octanoate, ethyl decanoate and diethyl butanedioate showed quite similar evolution patterns. These compounds showed a rapid increase in the first 5–7 days, while they tended to decrease thereafter and stagnated at the very end. The observed result agrees with that of Petropulos et al. [46], who reported that a further increase in maceration time led to a slight decrease in the ester concentration in red Vranec wines, possibly due to non-enzymatic hydrolysis. In general, maceration could influence the aroma composition by affecting the availability of different aroma precursors [47]. Indeed, in grapes, aroma components are present partly as free volatile forms and mainly as non-volatile precursors that are released during fermentation and aging of the wine or that are involved in the synthesis of the aroma [48]. Esters, both ethyl and acetate esters, are known to contribute significantly to the fresh and fruity aromas of wine. They are formed by yeast metabolism during alcoholic fermentation, namely, ethyl esters from fatty acid precursors and acetate esters from acetic acid and (higher) alcohols [49]. Moreover, these compounds are known to be synthetized during fermentation in concentrations that are usually higher than those theoretically expected based on their hydrolysis/synthesis equilibria [49], which explains a decrease in their concentrations during the prolonged maceration period. In addition, a reduction in some aroma compounds could also be due to their adsorption by certain macromolecules and skin components [47]. Finally, it has been reported that the presence of dissolved oxygen and unsaturated fatty acids significantly reduces the formation of acetate and ethyl esters during fermentation [50]. Lukić et al. [21] also suggested that an increased oxygen concentration may contribute to the reactions of oxidation and degradation of fermentation aroma precursors, which also leads to a lower concentration of aroma compounds in wine.

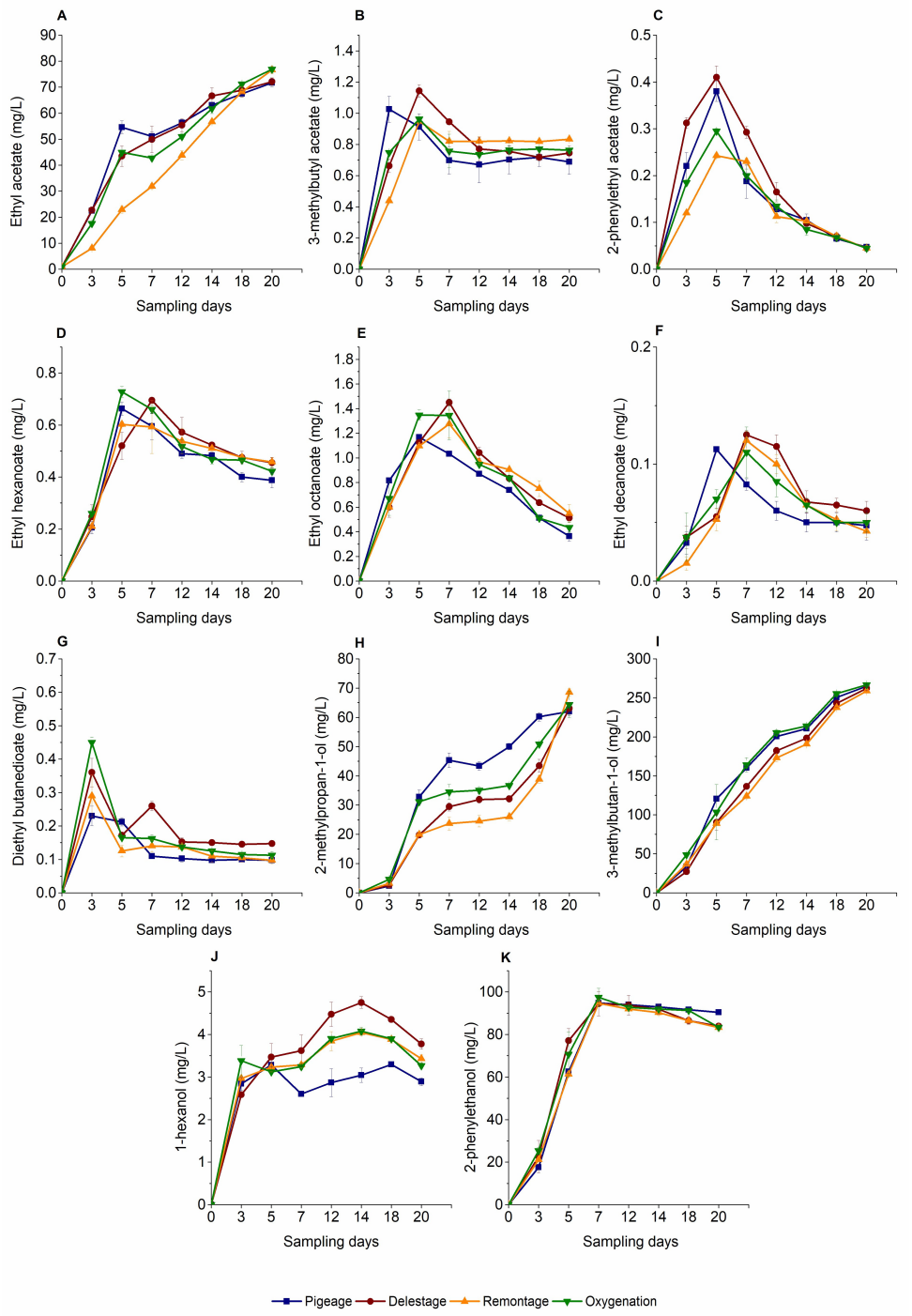


**Figure 3.** Changes in esters (**A**–**G**) and alcohols (**H**–**K**) of fermenting musts/wines during 20 days of maceration for studied winemaking treatments. Data presented as mean value of four repetitions ± standard deviation as error bars. ANOVA to compare data; different letters indicate statistical differences between musts/wines of all treatments at same sampling point (Tukey's test, $p < 0.05$).

Another important group of aroma compounds analyzed are the higher alcohols (Figure 3H–K), whose concentration also increased during the observed period, but in contrast to the esters, the differences between the treatments applied were statistically significant ($p < 0.05$). The highest concentrations of each alcohol in relation to the applied treatments were determined as follows: 2-phenylethanol in the control sample (pigeage), 1-hexanol in the délestage sample, 2-methylpropan-1-ol in the remontage sample, and 3-methylbutan-1-ol in the sample obtained by the oxygenation treatment. In addition, similar

increasing trends were observed for 2-methylpropan-1-ol and 3-methylbutan-1-ol during the maceration period, while 1-hexanol and 2-phenylethanol showed an increase in the first part of the maceration and a slight decrease thereafter. It is known that alcohols, especially higher alcohols, are formed by yeast, either directly from sugars or during amino acid catabolism via the Ehrlich pathway [1], and mainly contribute to fruity, floral and solvent-like aromas of wine [51]. At the beginning of fermentation, the amino acids are completely consumed by the yeasts, which later leads to the production of higher alcohols [50]. It is also suggested that the concentration of aroma compounds depends on the grape variety, the mechanical treatments applied (pump over, punch down, racking) and oxygen management during the maceration period [46,51,52]. For example, Cai et al. [51] showed that the pump over treatment at the end of alcoholic fermentation in pre-fermentative cold-macerated Cabernet Sauvignon resulted in a higher concentration of total higher alcohols, acetate and ethyl esters than the délestage treatment did. Additionally, Valero et al. [53] reported that the concentration of higher alcohols depends on the oxygenation conditions, with lower concentrations of these compounds being produced in the absence of oxygenation. In other words, the applied oxygenation promoted the synthesis of higher alcohols, which is consistent with the results of this study. In addition, Cai et al. [51] found that a higher concentration of dissolved oxygen in the must can lead to a greater formation of C6 aldehydes through the degradation of lipid acids, resulting in a higher concentration of C6 alcohols later during fermentation. The higher concentration of 1-hexanol in the délestage sample, as highlighted above, is good evidence of the greater importance of enzymatic oxidation. Moreover, a decrease in the C6 alcohol concentration as a function of the maceration time due to binding to macromolecules has been reported in the literature [46], which may have been the case for 1-hexanol in this study (Figure 3J). Nevertheless, it can be concluded that the application of different maceration treatments had a stronger influence on the extraction and polymerization of phenolic compounds in red Teran wines than on their aroma compounds.

### *3.3. Phenolic, Color and Aroma Composition of Wines after Pressing and Racking*

The trends in TP concentrations that were previously observed after 20 days of maceration were maintained after both pressing and racking (Table 2). This means that the remontage treatment contributed to the highest TP concentrations, while no significant differences were observed between the other three treatments (pigeage, délestage and oxygenation). In addition, the délestage treatment had significantly lower TA concentrations after pressing and racking ($p < 0.05$) compared to the other treatments. However, it is interesting to note that the TA loss was more pronounced in remontage than in pigeage or oxygenation, resulting in significantly lower concentrations in the former treatment than in the latter two ($p < 0.05$). The maceration technology strongly affects the wine's phenolic and sensory characteristics [3,5–7,9,14,34,44,45,54]. However, the grape variety and the initial composition of the grapes are decisive for the final impact of the maceration technology on the phenolic composition of the finished wine [43,44]. Therefore, the maceration technology must be selected according to the characteristics of the grape variety and the wine style. In addition, the phenolic differences between wines produced with different maceration techniques may be lost over time and during the further evolution of the wine [9,32,34].

**Table 2.** Total phenolics (TPs), total anthocyanins (TAs), chromatic characteristics (L*, a*, b*, C* and H*) and aroma compounds of Teran wines after pressing and racking for studied winemaking treatments.

| | Stage | Pigeage (C) | Délestage | Remontage | Oxygenation |
|---|---|---|---|---|---|
| | Pressing | | | | |
| Phenolics (mg/L) | TP | 2635.9 ± 20.8 $^{b}$ | 2607.3 ± 23.5 $^{b}$ | 2809.1 ± 27.3 $^{a}$ | 2661.5 ± 15.6 $^{b}$ |
| | TA | 617.8 ± 3.5 $^{a}$ | 512.7 ± 6.0 $^{b}$ | 610.2 ± 4.6 $^{a}$ | 615.2 ± 4.4 $^{a}$ |
| Color | L* | 10.88 ± 0.08 $^{b}$ | 12.87 ± 0.02 $^{a}$ | 11.27 ± 0.02 $^{b}$ | 10.87 ± 0.18 $^{b}$ |
| | a* | 39.00 ± 0.30 $^{b}$ | 41.35 ± 0.06 $^{a}$ | 39.78 ± 0.06 $^{b}$ | 40.12 ± 0.28 $^{b}$ |
| | b* | 16.84 ± 0.13 $^{bc}$ | 19.74 ± 0.03 $^{a}$ | 17.51 ± 0.02 $^{b}$ | 16.09 ± 0.29 $^{c}$ |
| | C* | 44.71 ± 0.33 $^{b}$ | 47.52 ± 0.06 $^{a}$ | 45.56 ± 0.06 $^{b}$ | 45.24 ± 0.12 $^{b}$ |
| | H* | 0.42 ± 0.00 $^{b}$ | 0.45 ± 0.00 $^{a}$ | 0.42 ± 0.00 $^{b}$ | 0.41 ± 0.01 $^{b}$ |
| | $\Delta E^*_{ab}$ | - | 4.2 | 1.1 | 1.3 |
| Aroma (mg/L) | Ethyl acetate | 71.35 ± 2.87 $^{b}$ | 72.13 ± 2.40 $^{ab}$ | 70.08 ± 2.03 $^{c}$ | 74.31 ± 4.36 $^{a}$ |
| | 3-methylbutyl acetate | 0.68 ± 0.01 $^{b}$ | 0.75 ± 0.07 $^{ab}$ | 0.85 ± 0.00 $^{a}$ | 0.77 ± 0.01 $^{ab}$ |
| | 2-phenylethyl acetate | 0.03 ± 0.01 $^{a}$ | 0.03 ± 0.01 $^{a}$ | 0.04 ± 0.00 $^{a}$ | 0.03 ± 0.01 $^{a}$ |
| | Ethyl hexanoate | 0.38 ± 0.01 $^{a}$ | 0.43 ± 0.02 $^{a}$ | 0.43 ± 0.04 $^{a}$ | 0.40 ± 0.00 $^{a}$ |
| | Ethyl octanoate | 0.32 ± 0.05 $^{a}$ | 0.40 ± 0.01 $^{a}$ | 0.34 ± 0.06 $^{a}$ | 0.37 ± 0.01 $^{a}$ |
| | Ethyl decanoate | 0.06 ± 0.01 $^{a}$ | 0.06 ± 0.00 $^{a}$ | 0.04 ± 0.00 $^{b}$ | 0.06 ± 0.01 $^{a}$ |
| | Diethyl butanedioate | 0.11 ± 0.02 $^{ab}$ | 0.16 ± 0.01 $^{a}$ | 0.10 ± 0.00 $^{b}$ | 0.11 ± 0.00 $^{ab}$ |
| | 2-methylpropan-1-ol | 66.32 ± 0.56 $^{a}$ | 67.23 ± 0.30 $^{a}$ | 62.05 ± 0.25 $^{b}$ | 61.50 ± 0.38 $^{b}$ |
| | 3-methylbutan-1-ol | 264.33 ± 0.57 $^{b}$ | 254.82 ± 1.07 $^{c}$ | 264.23 ± 0.07 $^{b}$ | 288.42 ± 1.26 $^{a}$ |
| | 1-hexanol | 2.80 ± 0.11 $^{c}$ | 3.65 ± 0.05 $^{a}$ | 3.19 ± 0.01 $^{b}$ | 3.19 ± 0.03 $^{b}$ |
| | 2-phenylethanol | 89.24 ± 0.81 $^{b}$ | 85.29 ± 1.16 $^{b}$ | 97.94 ± 2.70 $^{a}$ | 78.64 ± 0.84 $^{c}$ |
| | Racking | | | | |
| Phenolics (mg/L) | TP | 2575.9 ± 24.8 $^{b}$ | 2550.3 ± 27.9 $^{b}$ | 2745.1 ± 19.5 $^{a}$ | 2586.5 ± 13.2 $^{b}$ |
| | TA | 608.0 ± 2.2 $^{a}$ | 507.2 ± 5.0 $^{c}$ | 586.6 ± 2.9 $^{b}$ | 603.7 ± 2.8 $^{a}$ |
| Color | L* | 11.86 ± 0.03 $^{b}$ | 12.99 ± 0.02 $^{a}$ | 11.98 ± 0.33 $^{b}$ | 11.49 ± 0.04 $^{b}$ |
| | a* | 38.22 ± 0.11 $^{b}$ | 40.52 ± 0.06 $^{a}$ | 38.99 ± 0.59 $^{ab}$ | 39.32 ± 0.15 $^{ab}$ |
| | b* | 16.73 ± 0.05 $^{bc}$ | 19.91 ± 0.03 $^{a}$ | 17.37 ± 0.25 $^{b}$ | 16.32 ± 0.07 $^{c}$ |
| | C* | 44.81 ± 0.13 $^{b}$ | 47.48 ± 0.07 $^{a}$ | 45.84 ± 0.45 $^{b}$ | 45.36 ± 0.16 $^{b}$ |
| | H* | 0.43 ± 0.00 $^{ab}$ | 0.46 ± 0.00 $^{a}$ | 0.44 ± 0.01 $^{ab}$ | 0.41 ± 0.00 $^{b}$ |
| | $\Delta E^*_{ab}$ | - | 4.1 | 1.0 | 1.2 |
| Aroma (mg/L) | Ethyl acetate | 70.26 ± 2.73 $^{a}$ | 71.65 ± 2.77 $^{a}$ | 68.14 ± 2.40 $^{a}$ | 71.10 ± 0.23 $^{a}$ |
| | 3-methylbutyl acetate | 0.68 ± 0.05 $^{bc}$ | 0.70 ± 0.01 $^{ab}$ | 0.75 ± 0.07 $^{a}$ | 0.61 ± 0.07 $^{c}$ |
| | 2-phenylethyl acetate | 0.03 ± 0.01 $^{a}$ | 0.03 ± 0.01 $^{a}$ | 0.02 ± 0.01 $^{a}$ | 0.02 ± 0.01 $^{a}$ |
| | Ethyl hexanoate | 0.36 ± 0.04 $^{b}$ | 0.41 ± 0.01 $^{a}$ | 0.36 ± 0.03 $^{b}$ | 0.34 ± 0.00 $^{c}$ |
| | Ethyl octanoate | 0.34 ± 0.04 $^{a}$ | 0.35 ± 0.01 $^{a}$ | 0.30 ± 0.01 $^{b}$ | 0.32 ± 0.01 $^{b}$ |
| | Ethyl decanoate | 0.06 ± 0.01 $^{a}$ | 0.06 ± 0.01 $^{a}$ | 0.04 ± 0.01 $^{b}$ | 0.04 ± 0.01 $^{b}$ |
| | Diethyl butanedioate | 0.11 ± 0.01 $^{b}$ | 0.16 ± 0.01 $^{a}$ | 0.12 ± 0.01 $^{ab}$ | 0.11 ± 0.00 $^{b}$ |
| | 2-methylpropan-1-ol | 65.88 ± 1.98 $^{b}$ | 66.76 ± 1.34 $^{a}$ | 60.68 ± 1.08 $^{c}$ | 60.91 ± 2.22 $^{c}$ |
| | 3-methylbutan-1-ol | 268.11 ± 2.50 $^{b}$ | 259.88 ± 3.75 $^{c}$ | 265.49 ± 0.50 $^{b}$ | 279.27 ± 0.71 $^{a}$ |
| | 1-hexanol | 2.81 ± 0.01 $^{c}$ | 3.61 ± 0.06 $^{a}$ | 3.19 ± 0.06 $^{b}$ | 3.11 ± 0.09 $^{b}$ |
| | 2-phenylethanol | 87.78 ± 2.40 $^{b}$ | 82.61 ± 1.31 $^{b}$ | 92.40 ± 0.0 $^{a}$ | 74.61 ± 2.70 $^{c}$ |

Data presented as mean value of four repetitions ± standard deviation as error bars. ANOVA to compare data; different letters indicate statistical differences between musts/wines of all treatments at same time (Tukey's test, $p < 0.05$).

In addition, some differences in chromatic characteristics that were previously observed between the délestage and the other three treatments tended to decrease after pressing and especially after racking (Table 2). This primarily goes for the a* and H* values after racking, which only remained significantly higher for the délestage wine but not for the pigeage wine or the oxygenated wine. Nevertheless, the délestage wine showed significantly higher L*, b* and C* values after the pressing and racking than all the other wines did. This indicates that the délestage contributed to a brighter, slightly yellowish

but more vivid color, since higher C* values are associated with greater chromatic purity [34,44]. The results obtained are also consistent with the lower concentration of TAs found in the délestage samples and their further decrease, probably due to the formation of pyranoanthocyanins and, to a lesser extent, polymeric pigments. Namely, the color brightness/dullness is related to the concentration of anthocyanins, so that a decrease in anthocyanins usually leads to an increase in brightness. The accumulation of pyranoanthocyanins, which are formed by the direct reaction of anthocyanins and acetaldehyde, is also favored in the early stages of fermentation and under oxygenated conditions [17]. These compounds are known to contribute to the color change from red-purple to orange, as they have more red-yellow hues compared to anthocyanins [42]. It is also interesting to note that the oxygenated wine (introduced to higher doses oxygen) had significantly lower H* values compared to the délestage wine, in addition to significantly lower L*, b* and C* values, indicating that the wine was not overoxidized. In addition, analysis of the data for total color difference ($\Delta E^*_{ab}$) revealed that the highest difference from the control (pigeage) was obtained for the délestage treatment. Furthermore, the $\Delta E^*_{ab}$ values calculated after pressing and racking were around 4.0 CIELab units (Table 2), indicating that they were visually different from the control. A $\Delta E^*_{ab}$ threshold of 3 CIELab units has been shown to be sufficient to distinguish a wine poured into a wine glass with the human eye under the tasting conditions [55]. This further supports the importance of anthocyanin concentrations and the intensity and proportion of chemical changes associated with the copigmentation, polymerization and degradation of anthocyanins for the chromatic characteristics and color stability of wine [56].

Regarding aroma compounds, the trend observed during maceration continued after pressing and racking, with an additional decrease in the concentration of esters. Nevertheless, the results (Table 2) showed that there was no significant difference in the concentration of almost all the esters analyzed between the treatments studied. Only a significantly higher ($p < 0.05$) concentration of ethyl acetate was found in the wine produced by oxygenation after pressing, while this trend diminished after racking. In addition, the concentration of 3-methylbutyl acetate was significantly higher in the remontage sample after both pressing and racking. Cai et al. [51] reported that remontage influenced the aroma composition of Cabernet Sauvignon wine more effectively than punch down treatment during cold-maceration, resulting in an increase in acetate esters and a decrease in higher alcohols. On the other hand, the lowest concentrations of esters were obtained by the oxygenation treatment. Recently, Picariello et al. [17] investigated the effects of controlled oxygenation during the fermentative maceration of red Corvina must on the aroma compounds of the wines produced. The authors found that oxygenation resulted in wines with lower concentrations of ethyl and acetate esters, which is consistent with the results of this work. Similar results were also obtained in an earlier study, in which lower concentrations of esters were found in wines produced from oxygenated Pedro Ximenez must [53]. Generally, the decrease in esters in young wines during maturation and storage is mainly due to two mechanisms: hydrolysis and, to a lesser extent, the oxidation process [57].

The differences in the concentration of the higher alcohols established during maceration changed slightly after pressing and racking. Significantly higher ($p < 0.05$) concentrations of 2-methylpropan-1-ol and 1-hexanol were found in the délestage samples, while the oxygenation treatment resulted in wines with higher concentrations of 3-methylbutan-1-ol compared to the other three treatments. In addition, the concentration of 2-phenylethanol, the most desired higher alcohol in wines due to its positive flowery, rose nuances [1], was highest in the remontage sample.

Generally, these compounds are formed during fermentation and can reach concentrations in the range of 150–550 mg/L. Moreover, their cumulative concentration below 300 mg/L could contribute to the complexity of a wine's aroma, while a concentration above 400 mg/L could have a negative effect [1], but a later sensory analysis will show that the negative effect was not pronounced in the wines produced. In general, their concentration in wines depends on the concentration of amino acids (valine, leucine, isoleucine,

threonine and phenylalanine) in the must (catabolic synthesis), but they are also produced de novo from a sugar substrate (anabolic pathway) [49]. In their review, the same authors emphasized the importance of amino acids for the formation of higher alcohols: they contribute to the total nitrogen levels of the must, and the amount of higher alcohols that are formed from the carbohydrates (anabolic) depends strongly on the nitrogen levels. They also reported that both catabolic and anabolic synthesis can be reduced by the addition of nitrogen. Therefore, the potentially low total nitrogen level in the Teran must could be the reason for the elevated concentrations of higher alcohols in the final wines, despite the treatments applied. Nevertheless, some authors reported the importance of cap management treatments. As already mentioned for esters, Cai et al. [51] found that the aroma of Cabernet Sauvignon wine was strongly affected by the applied treatment, with only pumping over leading to a decrease in certain alcohols and an increase in some esters. On the other hand, Fischer et al. [13] observed a very small and inconsistent impact on the aroma composition of German red wines of different varieties after the application of pump over and punch down treatments.

### *3.4. Sensory Characterization of Produced Wines by Pigeage, Délestage, Remontage and Oxygenation Treatments*

All the tested wines were graded by high scores according to the 100-point evaluation method as follows: remontage (86.1 points) > délestage (83.5 points) > pigeage (82 points) > oxygenation (80 points). From the results given, it can be seen that the remontage sample received the highest score, while oxygenation resulted in the wine with the lowest score. The results of the QDA of the wines produced are shown in Figure 4. As can be seen, the treatments applied resulted in different sensory profiles of the Teran wines. The control treatment (pigeage) resulted in a wine with intensive aromas of sour cherry, blackcurrant and redcurrant, as well as an intensive astringency intensity and a more pronounced body. The délestage treatment resulted in a wine with moderate intensity of sour cherry, cherry, blueberry and plum flavors, along with intensive body roundness and moderate astringency intensity. Furthermore, the wine with the highest intensities of raspberry, blackberry and compote notes, moderate intensity of sour cherry and plum, along with moderate notes of blackcurrant and cherry, was produced by the remontage treatment. In addition, this treatment also resulted in a very intensive astringency perception and an intensive body sensation, which is consistent with the highest TP concentration determined in this sample. Finally, the wine produced by oxygenation showed the lowest intensities of astringency and body sensation, together with an intensive sour cherry flavor. Besides these above-stated characteristics, this wine was characterized by a slight intensity of spicy/herbs and forest fruit aromas. There are only a few studies that deal with the effect of maceration or oxygenation treatments on the sensory properties of the wines produced. Cai et al. [51], for example, demonstrated that the fruity aromas in the wines produced by pumping over were intensified and the chemical aromas minimized compared to the punch down treatment. A recent study by Lukić et al. [21] showed that oxygenation resulted in wines with lower scores of fruity–flowery aromas (due to the absence of fermentation esters), while the green aroma (C6 alcohols) was increased. Moreover, it has already been highlighted that excessive oxidation leads to unfavorable changes in aroma, especially the loss of fruity notes [19]. It is worth mentioning that sour cherry is considered a main descriptor of Teran wine [58], and among the wines produced, the highest intensity of this aroma was found in wines produced with the control (pigeage) treatment, followed by oxygenation and remontage, while the lowest intensity was found in the délestage treatment. In general, the intensity of astringency decreases with the aging of red wines due to the structural changes in polyphenols, while the persistence of the wine's aroma increases [59]. Indeed, older red wines are characterized by lower concentrations of various low-molecular-weight phenolic compounds and anthocyanins and higher concentrations of polymeric pigments, while younger wines have higher concentrations of anthocyanins and other phenolic compounds [60]. Thus, oxygenation during these initial stages of winemaking could

potentially accelerate the structural changes in polyphenols (e.g., polymerization and condensation reactions), resulting in a wine with a lower intensity of astringency.

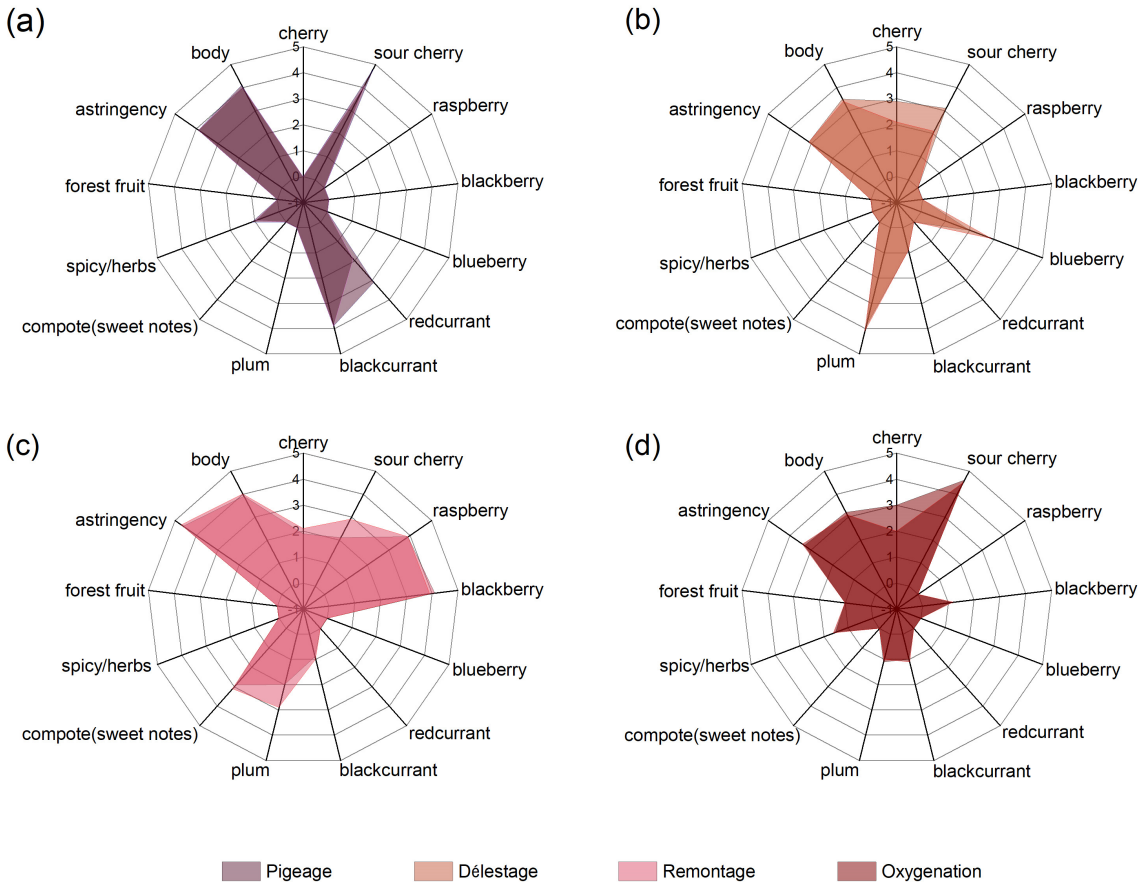


**Figure 4.** Quantitative descriptive analysis (QDA) of produced Teran wines: pigeage (**a**); délestage (**b**); remontage (**c**); and oxygenation (**d**).

## 4. Conclusions

The application of various treatments (pigeage, délestage, remontage and oxygenation) during the prolonged maceration of Teran wine primarily affected the phenolic compound contents and chromatic characteristics and, to a lesser extent, the aroma compounds. Remontage (pump over) contributed to significantly higher concentrations of total phenols, while délestage after 20 days of maceration resulted in significantly lower concentrations of total phenols and total anthocyanins. Moreover, both trends were maintained even after pressing and racking. In addition, the chromatic characteristics of the délestage wine differed significantly from those of the other treatments due to the higher L*, b* and C* values. Thus, a lighter, slightly yellowish but more "vivid" color was obtained by the délestage treatment of Teran wine. Moreover, only slight changes in aroma composition were observed between treatments during the maceration period, especially for the higher alcohols, while the concentration of esters was not affected. The differences in aroma composition were more pronounced after pressing and racking, with the délestage treatment resulting in the highest concentrations of most of the esters analyzed and remontage in the highest concentrations of desirable 3-methylbutyl acetate and 2-phenylethanol, while oxygenation led to the highest concentrations of 3-methylbutan-1-ol. Finally, the sensory evaluation of the Teran wines showed that the wine obtained by remontage had the best overall quality, with a well-structured body, the highest intensity of raspberry, blackberry and compote notes, a moderate intensity of sour cherry and plum, and less pronounced blackcurrant and cherry notes.

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